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Efficiency of cleaning agents in fouling removal from ceramic membranes after filtration of municipal digestate liquid fraction

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Abstract: The liquid fraction of digestate, an important byproduct of anaerobic digestion in biogas plants treating municipal waste, has a complex and variable chemical composition and contains significant impurities of various types. Effective treatment of this fraction prior to further use poses a technological challenge, particularly in light of increasingly stringent environmental standards. Pressure-driven membrane processes, especially those using flat ceramic membranes, have the potential to efficiently separate contaminants and recover valuable components and water; however, they are prone to severe fouling. This study evaluated the effectiveness of selected chemical reagents for cleaning flat ceramic membranes after filtration of the liquid fraction of digestate from a municipal biogas plant. The results revealed that the porous structure of ceramic membranes significantly influences their transport properties, fouling mechanisms, and regeneration efficiency. Ultrafiltration membranes with molecular weight cut-offs of 5 or 15 kDa provided an optimal balance between separation efficiency, fouling resistance, and chemical cleaning efficiency. Additionally, fouling control effectiveness strongly depended on matching regeneration strategies to membrane characteristics and sludge type.

Introduction

The liquid fraction of digestate is a significant byproduct of the anaerobic digestion process conducted in biogas plants, particularly those relying on municipal waste as a substrate (Tuszynska et al., 2021). Given its physicochemical properties and chemical composition, it is gaining increasing importance in the context of digestate management and sustainable resource utilization (Świątczak et al., 2019). This fraction contains substantial quantities of nitrogen, phosphorus, potassium, and organic matter in readily digestible forms, rendering it a promising organic fertilizer or a source of components for recovery (Li et al., 2016). Concurrently, the presence of organic, microbial contaminants and trace amounts of micropollutants, including pharmaceuticals, heavy metals, and microplastics, poses significant environmental and technological challenges (Porterfield et al., 2023).

In biogas plants processing the organic fraction of municipal waste (BIO), the liquid digestate is characterized by greater compositional heterogeneity and a potentially higher pollutant load compared to digestate derived from agricultural substrates (Ali et al., 2019). The composition and properties of the liquid fraction of digestate depend on several factors, including the type of feedstock used, fermentation process conditions, and the phase separation technology employed (Monfet et al., 2018). Effective management of these systems is currently a major challenge for biogas plant operators, especially in light of increasingly stringent environmental regulations and the need to minimize impact on water and soil environments (Paolini et al., 2018).

Due to the high concentrations of nutrients, organic matter, and mineral salts, as well as impurities that are difficult to remove, treatment of the liquid fraction of digestate is required prior to its use or discharge (Porras-Socias et al., 2024). A variety of purification methods are employed in practice, encompassing mechanical (e.g., flotation, sedimentation), chemical (e.g., precipitation), and biological (e.g., nitrification-denitrification) processes (Chojnacka & Moustakas, 2024). While these techniques can effectively remove selected groups of contaminants, they often do not guarantee the production of liquids of sufficient quality, particularly in the context of increasingly stringent environmental standards and water reuse requirements (Ferri & Bolelli, 2025).

In recent years, pressure-driven membrane processes, including microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO), have gained considerable popularity (Shannon et al., 2008). These processes offer significant potential for the efficient separation of suspended solids, organic matter, biogens, and micropollutants (Alonso et al., 2024; Matter, 2024). The effectiveness is well established, and their ability to integrate with other technologies further enhances their applicability. Such integration enables the purification of digestate liquid to a quality suitable for water recirculation, as well as the concentration and recovery of valuable components (Proskynitopoulou et al., 2024).

Of particular interest is the use of flat ceramic membranes. These materials exhibit high chemical, mechanical, and thermal resistance, contributing to enhanced durability and operational stability under the severe fouling conditions characteristic of digestion liquids (Asif & Zhang, 2021). In comparison to polymeric membranes, ceramic membranes demonstrate greater resistance to aggressive cleaning agents and a prolonged operational lifespan (Gitis & Rothenberg, 2016). Despite these advantages, the use of flat ceramic membranes for treating the liquid fraction of digestate, especially from municipal biogas plants, remains under-explored and sparsely represented in the scientific literature.

One of the primary technological limitations associated with the use of membranes for digestate liquid treatment is fouling (Zielińska & Bułkowska, 2025), defined as the clogging or blockage of membrane pores by deposited particles. Fouling is commonly classified according to the type of substances responsible for its occurrence. Four main types are distinguished: colloidal fouling, organic fouling, biofouling, and mineral fouling, also referred to as scaling (Gul et al., 2021). The consequences of fouling include reduced permeate flux, deterioration of permeate quality, and increased operational costs resulting from frequent membrane cleansing and shortened membrane lifespan.

Membrane fouling mechanisms can be categorized into several basic types: standard blockage, involving the gradual narrowing of pore diameters due to particle deposition; complete blockage, characterized by permanent pore closure by particles comparable in size to the pore diameter; intermediate (medium) blockage, associated with a partial restriction of flow within the membrane structure; and the formation of a surface deposit or cake layer, which acts as an additional resistance to liquid transport (Pereira et al., 2023). Differences among these mechanisms determine the reversibility of fouling and the effectiveness of specific membrane regeneration strategies (Gul et al., 2021).

Notably, digestate liquid derived from municipal waste contains high concentrations of organic matter and surfactants, which can lead to severe fouling (Meng et al., 2023; Wang et al., 2023). This phenomenon necessitates the implementation of specialized pretreatment methods and the application of chemical and hydraulic cleaning procedures for membrane filtration.

An important factor in reducing fouling intensity is the appropriate pretreatment of raw water prior to the target membrane process (Poirier et al., 2023). Pretreatment methods include coagulation, sedimentation, filtration, oxidation and adsorption, among other things (Arhin et al., 2016). These processes enable the removal of suspended solids, colloids and a portion of organic matter. In multi-stage systems, microfiltration membranes can also serve as a pretreatment step, significantly reducing the pollutant load (e.g., before NF or RO). Effective pretreatment of the raw material reduces membrane loading, extends membrane service life, and stabilizes system operation (Ahmed et al., 2023).

In global operating practice, the prevailing approach to mitigating fouling involves the use of chemical cleaning methods for membrane surfaces (Goh et al., 2018). These treatments are an indispensable component of maintaining membrane system performance and are essential for ensuring stable, long-term plant operation (Filloux et al., 2015). The selection of cleaning chemicals employed in this process is dependent upon the type of sludge in question. Alkaline solutions, such as NaOH, are commonly used for the removal of organic matter (Dow et al., 2017). Inorganic fouling is addressed using acidic solutions, including citric acid and HCl (Gryta, 2008), while oxidizing agents such as sodium hypochlorite are employed to eliminate biofilm and microorganisms (Stewart et al., 2001). Chemical cleaning of membranes is typically performed cyclically, often in an automated manner, ensuring sustained cleaning efficiency while minimizing the risk of membrane damage (Kazemimoghadam & Mohammadi, 2007).

In addition to selecting suitable cleaning agents, it is essential to define cleaning parameters tailored to the specific fouling type and the characteristics of the digestate liquid. Frequent cleaning increases operating costs and accelerates membrane degradation, whereas insufficient cleaning may result in irreversible permeability loss and deterioration of permeate quality (Das et al., 2022). Consequently, the development of comprehensive fouling management strategies, including online monitoring (e.g., measuring transmembrane pressure drop and flux changes), is crucial for optimizing membrane system operation (Maddah & Chogle, 2017).

The scientific literature extensively addresses issues related to the cleaning of polymer membranes in both water and wastewater treatment. However, far less attention has been given to the cleaning of ceramic membranes, despite their growing technological importance and high chemical resistance (Kang & Ge, 2025). Previous research has mainly focused on the use of membranes to treat liquid agricultural waste, while the treatment of municipal digestate is a relatively unexplored area (Urbanowska & Kabsch-Korbutowicz, 2021, 2023). Given that ceramic membranes are highly effective at removing contaminants from such streams, it is also necessary to develop effective regeneration strategies to fully exploit their practical potential.

In response to these challenges, the present study aimed to evaluate the effectiveness of various chemical reactants in cleaning flat ceramic membranes fouled during filtration of the liquid fraction of digestate from a municipal waste biogas plant. The novelty of this study lies not only in the uncommon application of flat ceramic membranes for municipal biogas plant digestate treatment, but also in the focus on developing chemical cleaning strategies that are effective, safe, and economical. The findings of this study may serve as a foundation for optimizing operational procedures for these membrane systems and for the broader implementation of ceramic membranes in challenging waste management applications.

Materials and research methods

The present study utilizes six commercially available flat ceramic membranes made of titanium oxide (TiO_2), zirconium oxide (ZrO_2), or a mixture thereof (ZrO_2-TiO_2). The membranes differ in their cut-off values and are all manufactured by Tami Industries (France). The membranes used in this study are characterized by a typical asymmetric structure consisting of a thin separation layer responsible for effective filtration and a thicker support layer that provides mechanical stability and facilitates the transport of the purified medium. A comprehensive summary of the membrane's characteristics is provided in Table 1.

The liquid digestate fraction obtained from the digestion of the organic fraction of municipal waste at a biogas plant operating at one of the waste management facilities in Lower Silesia Province, Poland (50°53'15.5 "N, 17°23'28.0 "E) was investigated. The fraction was separated from the digestate pulp using sedimentation centrifuges. The characteristics of the analyzed fraction are presented in Table 2.

Four types of chemical reactants were used to investigate the cleaning efficacy of flat ceramic membranes: sodium hydroxide (NaOH) solutions at concentrations of 0.0025M and 0.01M, hydrochloric acid (HCl) solutions at pH 3 and 4, citric acid solutions at the same pH values, and the anionic surfactant sodium dodecyl sulfate (SDS) at concentrations of 1 g/dm³ and 5 g/dm³. Supplementary membrane cleaning was performed using distilled water.

A series of experiments was conducted to assess the cleaning efficiency of flat ceramic membranes. The experimental setup, shown in Figure 2, comprised a test rig equipped with a Sterlitech laboratory unit containing a 3.8 dm³ Type 316 stainless steel pressure chamber, which enabled processes employing flat ceramic membranes to be performed. The system operated in dead-end mode at a constant transmembrane pressure of 0.2 MPa.

Prior to testing, all flat ceramic membranes were subjected to a pretreatment procedure. The pretreatment consisted of alkaline cleaning in a sodium hydroxide (NaOH) solution at a concentration of 15-20 g/dm³ at 80°C for 30 minutes, followed by rinsing with water until a neutral pH was achieved. Subsequently, an acid cleaning step was performed and likewise followed by rinsing to neutral pH. For micro- and ultrafiltration membranes, acid cleaning was performed using

Table 1. Characteristics of the membranes used in the experiments based on (Sterlitech, 2024) and own research

Membrane type	**Ceramic Flat Sheet Membrane**					
	Fine UF	**Fine UF**	**UF**	**UF**	**MF**	**MF**
cut-off	1 kDa	5 kDa	15 kDa	50 kDa	0.14 µm	0.45 µm
Active layer	TiO_2	TiO_2	ZrO_2	ZrO_2	ZrO_2-TiO_2	ZrO_2-TiO_2
Contact angle,	59.6	57.6	43.8	42.4	36.6	36.7
Average pore size, nm	35.53	37.69	52.45	67.09	122	290
pH range	2-14	2-14	0-14	0-14	0-14	0-14
Filtration area, cm²	56					
Nominal thickness, mm	2.5					
Max. pressure, MPa	0.4					
Max temp., °C	350					

Fig 1. Example photograph of the 5 kDa cut-off flat ceramic membrane used in this study (a) and its SEM image (1000x magnification) (b)

Table 2. Physicochemical and microbiological characteristics of the municipal digestate liquid fraction

Examined Parameter	Value	Analytical method used for parameter determination	Apparatus	Standard or documented testing procedure
pH	6.5	Potentiometric method	Digital multimeter HQ40D with IntelliCALTM PHC 101 electrode	PN-EN ISO 10523:2012
Conductivity, mS/cm	21.4	Conductometric method		PN-EN 27888:1999
Total suspended solids, mg/dm^3	55 230	Weight-based method	-	PN-78 C-04541
COD mg O_2/dm^3	6 990	Bichromate method	-	Standard Methods 5220 D
BOD_5, mg O_2/dm^3	2 770	Dilution method	-	PN-EN 1899-1:2002
DOC, mg C/dm^3	3 995	NPOC high temperature oxidation method; thermal method	Hach IL550 carbon analyzer (Hach, Ames, IA, USA)	PN–EN 1484:1999
$N\text{-}NH_4^+$, mg/dm^3	856	Spectrophotometric method, direct nesslerisation	Shimadzu UV-VIS 1800 (Shimadzu Corporation, Kyoto, Japan)	Standard Methods 4500-NH3 C
$N\text{-}NO_2^-$, mg/dm^3	6.11	Ion chromatography method	Thermo Scientific Dionex Aquion ion chromatograph with a conductometric detector for anions or cations analysis (Thermo Fisher Scientific, Waltham, MA, USA)	PN-EN ISO 10304-2001
$N\text{-}NO_3^-$, mg/dm^3	below the limit of detection			
PO_4^{3-}, mg/dm^3	18.4	Spectrophotometric method with ascorbic acid	Shimadzu UV-VIS 1800 (Shimadzu Corporation, Kyoto, Japan)	PN-EN ISO 3946:2000
SO_4^{2-}, mg/dm^3	38	Ion chromatography method	Thermo Scientific Dionex Aquion ion chromatograph with a conductometric detector for anions or cations analysis (Thermo Fisher Scientific, Waltham, MA, USA)	PN-EN ISO 10304-2:2001
Na, mg/dm^3	1 850	Ion chromatography method	Thermo Scientific Dionex Aquion ion chromatograph with a conductometric detector for anions or cations analysis (Thermo Fisher Scientific, Waltham, MA, USA)	PN-ISO 14911:(2001)
K, mg/dm^3	1 760			
Ca, mg/dm^3	389	Titration method	-	PN-ISO 6058:1999
Mg, mg/dm^3	231	Titration method	-	PN-C 04554-4:1999

Examined Parameter	Value	Analytical method used for parameter determination	Apparatus	Standard or documented testing procedure
Cl^-, mg/dm^3	2 249	Ion chromatography method	Thermo Scientific Dionex Aquion ion chromatograph with a conductometric detector for anions or cations analysis (Thermo Fisher Scientific, Waltham, MA, USA)	PN-EN ISO 10304-2:2001
Fe, mg/dm^3	2.8	Spectrophotometric method	Shimadzu UV-VIS 1800 (Shimadzu Corporation, Kyoto, Japan)	PN-ISO 6332:2001
Mn, mg/dm^3	3.9			PN-92/C-04590/03
Cu, mg/dm^3	0.098	Atomic absorption spectroscopy (ASA) with flame atomization	Atomic absorption spectrometer iCE 3500 (Thermo Fisher Scientific, Waltham, MA, USA)	PN-ISO 8288:2002
Zn, mg/dm^3	0.633			
Li, mg/dm^3	6.3			
Co, mg/dm^3	0.138			
Ni, mg/dm^3	0.261			
Hg, mg/dm^3	0.0036	Atomic absorption spectroscopy (ASA) - selective for Hg with concentration by amalgamation	AMA 254 mercury analyzer (Leco Corporation, St. Joseph, MI, USA)	PN-EN ISO 12846:2012
Ba, mg/dm^3	0.246	Atomic absorption spectroscopy (ASA) with flameless atomisation	Atomic absorption spectrometer iCE 3500 (Thermo Fisher Scientific, Waltham, MA, USA)	PN-EN ISO 11885:2009
As, mg/dm^3	0.0005			
Cr, mg/dm^3	0.229	Atomic absorption spectroscopy (ASA) with flame atomisation and flameless atomisation	Atomic absorption spectrometer iCE 3500 (Thermo Fisher Scientific, Waltham, MA, USA)	PN-EN 1233:2000
Pb, mg/dm^3	0.027	Atomic absorption spectroscopy (ASA) with flame atomisation and flameless atomisation	Atomic absorption spectrometer iCE 3500 (Thermo Fisher Scientific, Waltham, MA, USA)	PN-ISO 8288:2002
Cd, mg/dm^3	0.001			
mesophilic bacteria, CFU/cm^3	$111 \cdot 10^6$	In-depth culture	-	PN-EN ISO 8199
thermophilic bacteria, CFU/cm^3	$163 \cdot 10^2$			

either a 58% nitric acid (HNO_3) or a 75% phosphoric acid (H_3PO_4) solution at a concentration of 5 cm^3/dm^3, at 50°C for 15 minutes. In the case of fine ultrafiltration (UF) membranes, only a 75% H_3PO_4 solution at a concentration of 1 cm^3/dm^3 was used, in accordance with the manufacturer's guidelines, also at 50°C for 15 minutes.

To induce and assess membrane fouling, the liquid digestate fraction was continuously filtered through the membrane. Fouling was defined as the point at which the permeate flux (J_f) stabilized and no further measurable change was observed. Subsequently, a cleaning step was performed by replacing the feed solution with an appropriate cleaning agent. All processes were conducted at a constant transmembrane pressure (TMP) of 0.2 MPa until the permeate flux stabilized or recovered to its initial value (J_0).

The membrane susceptibility to fouling was evaluated on the basis of relative permeability, expressed as the ratio of permeate flux during cleaning (J) to the initial permeate flux (J_0). The cleaning efficiency (η) was defined as the percentage recovery of the initial permeate flux and was calculated according to the following equation:

$$\eta = [1 - (J_0 - J) / (J_0 - J_f)] \cdot 100\%$$

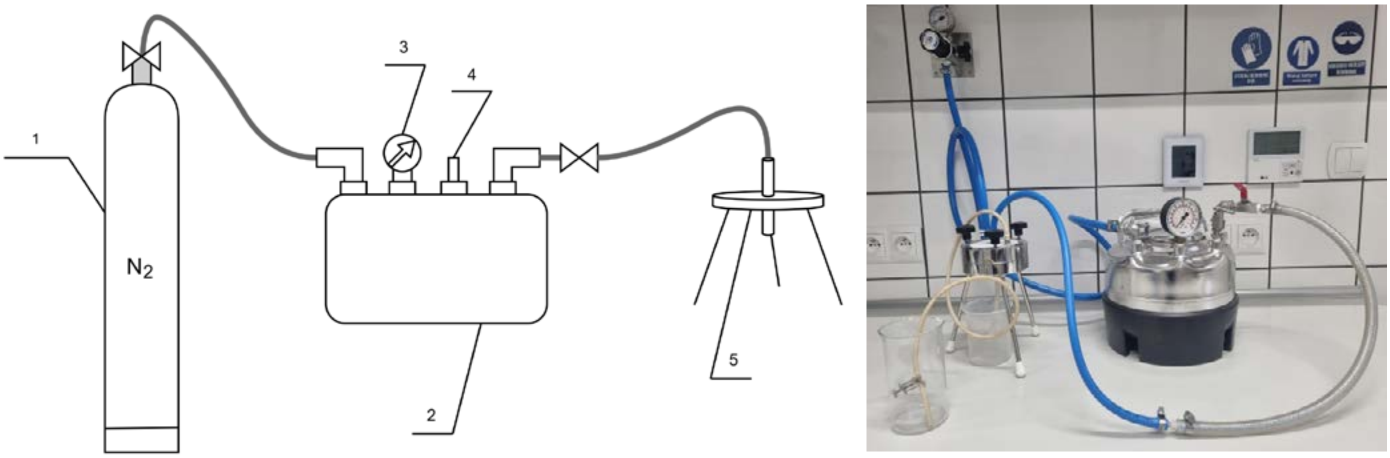

Fig 2. Schematic of Sterlitech laboratory installation (1 - compressed nitrogen cylinder, 2 - pressure vessel, 3 - pressure gauge, 4 - safety valve, 5 - ceramic membrane in casing)

Membrane susceptibility to fouling was also assessed based on the membrane resistance R_{mem}, calculated as:

$$R_{mem} = TMP / (\mu \cdot J),\ m^{-1}$$

All permeate flux values were determined by measuring the volume of permeate collected over a period of 1 minute.

Results

Figure 3 shows the transport properties of flat ceramic membranes with different pore diameters. The process comprised three steps: membrane conditioning; fouling with a solution of the liquid fraction of municipal waste digestate; and chemical cleaning using a 0.01M NaOH solution. Final filtration was performed using distilled water. The total membrane resistance values were correlated with pore size: membranes with larger pores (e.g., Ceram 0.45 µm with an average pore diameter of 290 nm) exhibited the lowest initial resistance (~5.96×10^{10} m^{-1}), whereas membranes with smaller pores (e.g., Ceram 1 kDa with an average pore diameter of ~35 nm) showed significantly higher initial resistance (~1.08×10^{12} m^{-1}).

All membranes exhibited a similar pattern of resistance changes throughout the process. During the conditioning stage, membrane resistance remained stable. This was followed by a sharp increase in resistance during the first 20 minutes of fouling, attributed to intense adsorption and pore blocking. Subsequently, resistance stabilized after approximately 120 minutes at a quasi-steady level. This behavior corresponds to the two-stage fouling mechanism described in the literature, consisting of initial pore blocking (standard and intermediate) followed by the formation of a surface foulant layer (cake layer) composed primarily of organic compounds. As reported in previous studies, smaller molecules, including dissolved organic compounds, are particularly prone to penetrating the interior of membrane pores, leading to irreversible fouling, which is characteristic of low-cut-off membranes.

The cleaning stage resulted in a significant reduction in membrane resistance, with the most rapid decrease observed during the first few minutes, after which a new stable level was reached. Microporous membranes (Ceram 0.14 µm and 0.45 µm) exhibited the highest cleaning efficiency, achieving complete permeate flux recovery (100%). In contrast, membranes with smaller pore sizes showed only partial recovery, indicating the presence of membrane-blocking compounds that are difficult to remove chemically. Specifically, permeate flux recovery reached 82% for the Ceram 50 kDa membrane and 97% for the Ceram 15 kDa membrane. According to a literature review by Bodzek and Płatkowska (2009), the most common difficult-to-remove foulants include proteins, amino sugars, polysaccharides, and poly(hydroxy-aromatic) compounds, which may adsorb onto the membrane surface or penetrate its structure.

It was also observed that membranes with smaller pores, particularly those with cut-off values in the range of 1-15 kDa, were more susceptible to irreversible fouling. This observation is consistent with fouling mechanisms described in the literature as standard and complete pore blocking. In contrast, microporous membranes (0.14 and 0.45 µm) were primarily affected by surface fouling mechanisms, such as gel or cake layer formation. This type of fouling is largely reversible and results in full recovery of transport properties after cleaning. Such behavior corresponds to fouling mechanisms associated with concentration polarization and gel accumulation of organic compounds, which are typical of solutes larger than the membrane pores.

The results indicate that membranes with larger average pore sizes exhibit lower total resistance and are more prone to reversible surface fouling. Conversely, membranes with smaller pores are more susceptible to irreversible internal fouling, commonly referred to as penetration fouling. Literature data confirm that this type of fouling is more difficult to remove, especially in the presence of hydrophilic colloids and dissolved organic matter fractions smaller than 0.5 kDa.

Accordingly, the observed trends in membrane resistance during the filtration process are consistent with the membranes' porous structure and the fouling mechanisms described in the literature. Membrane selection should therefore consider not only separation performance but also fouling resistance and the ease of regeneration. For practical applications where efficient regeneration and low risk of permanent loss of transport properties are priorities, microporous membranes may be preferable. Conversely, low cut-off membranes can provide more efficient separation of fine particles but require more advanced cleaning procedures and feedwater pretreatment strategies.

Analysis of the decrease in relative flux (J/J_0) during the filtration process (Figure 4) revealed that all of tested membranes experienced rapid fouling. The J/J_0 values were already low (<0.08) at the beginning of the process and decreased further over time. Membranes with a cut-off values of 5 or 15 kDa were the least susceptible to fouling, as their J/J_0 values remained higher throughout the experiment. Notably, the 5 kDa membrane exhibited the smallest flux reduction, indicating an optimal balance between separation performance and fouling resistance.

A significant reduction in permeability was observed for membranes with extreme pore sizes — both very compact (1 kDa) and large (0.14 µm and 0.45 µm). Microfiltration membranes (0.14 µm and 0.45 µm) exhibited the highest degree of fouling, with J/J_0 values around 0.01 at the beginning of the process, dropping below 0.001 after 180 minutes. This sharp decline in permeability likely reflects intensive particle deposition and the formation of a dense cake layer on the membrane surface.

The results suggest that, for applications requiring long-term operation without frequent cleaning, ultrafiltration membranes with medium cut-off values of 5-15 kDa are the most favorable. This finding is particularly relevant for wastewater treatment and water recovery, where the presence of colloids and macromolecular substances can rapidly clog membranes with pores that are either too large or too small.

Figure 4. Relative permeability of the membrane for liquid municipal digestate during membrane fouling

Figure 5 shows significant differences in chemical cleaning efficiency depending on the type of agent used and the type of membrane when analyzing the effectiveness of membrane regeneration after filtration of the liquid fraction of municipal digestate. The best results were achieved using a 0.01M NaOH solution, which fully restored the flux of four of the six tested membranes to the initial level ($J/J_0 = 1$). For the remaining two membranes (5 and 50 kDa), the J/J_0 value was 0.98. The high efficiency of this agent suggests that the predominant fouling mechanism was the deposition of acidic organic matter or matter prone to degradation in an alkaline environment. In contrast, an HCl solution at pH 3 had a high J/J_0 efficiency of ≥ 0.95 for most membranes. This may indicate the presence of an inorganic fraction, such as metal precipitates or calcium compounds, that is susceptible to dissolution in an acidic environment.

The effectiveness of chemical cleaning can be ranked in the following order:
NaOH 0.01M > HCl pH 3 > NaOH 2.5mM > HCl pH 4 > citric acid pH 3 > citric acid pH 4 > SDS 5% > SDS 1% > distilled water.

Distilled water demonstrated the lowest efficiency, particularly for membranes with larger pore sizes. In these cases, the J/J_0 value did not exceed 0.34 after cleaning and often remained below 0.2. However, pure water was found to be relatively effective in regenerating membranes with a low molecular weight cut-off (e.g., 1 kDa), which may reflect a lower proportion of impurities permanently bound to the membrane surface.

Thus, the highest cleaning efficiency was observed for alkaline and acidic agents at low pH and moderate concentrations, whereas, physical cleaning (i.e., distilled water) was ineffective. These results confirm the necessity of chemical cleaning strategies for membrane processes involving the liquid fraction of municipal digestate.

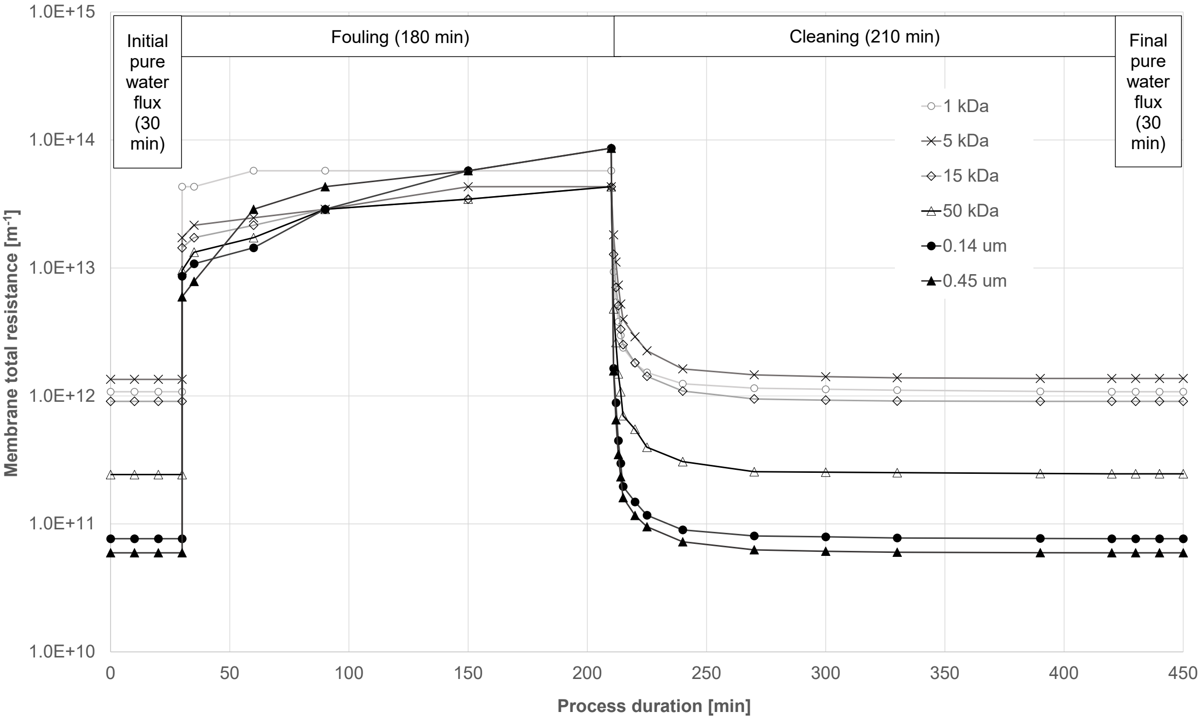

Fig 3. Total membrane resistance for various flat ceramic membranes fouled with liquid fraction of municipal origin digestate and treated with 0.01M NaOH

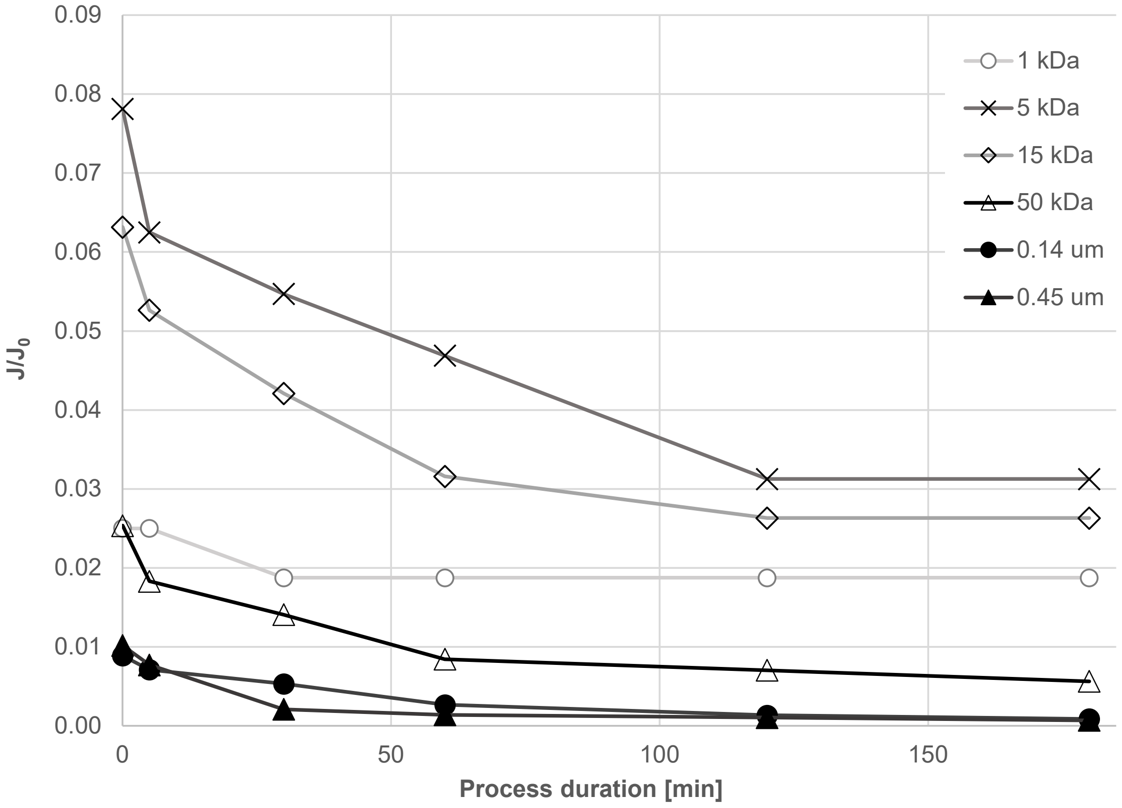

Fig 4. Relative permeability of the membrane for liquid municipal digestate during membrane fouling

Figure 6 shows the percentage purification efficiency after a 3-hour membrane regeneration process. Significant differences in cleaning efficiency were observed depending on the membrane characteristics (microfiltration and ultrafiltration) and the cleaning agent used. The highest cleaning efficiency was obtained with a 0.01M NaOH solution. For most of the tested membranes, especially ultrafiltration membranes, this solution enabled the recovery of nearly the entire original flux (efficiency close to 100%). Comparable results were obtained with an HCl solution at pH 3, indicating that the accumulated sludge on the membranes contained both organic components soluble in an alkaline environment and inorganic components that dissolve in an acidic environment.

Weaker agents were less effective. For example, the cleaning efficiencies of NaOH (2.5mM), HCl (pH 4), and citric acid (pH 4) were significantly lower, especially for membranes

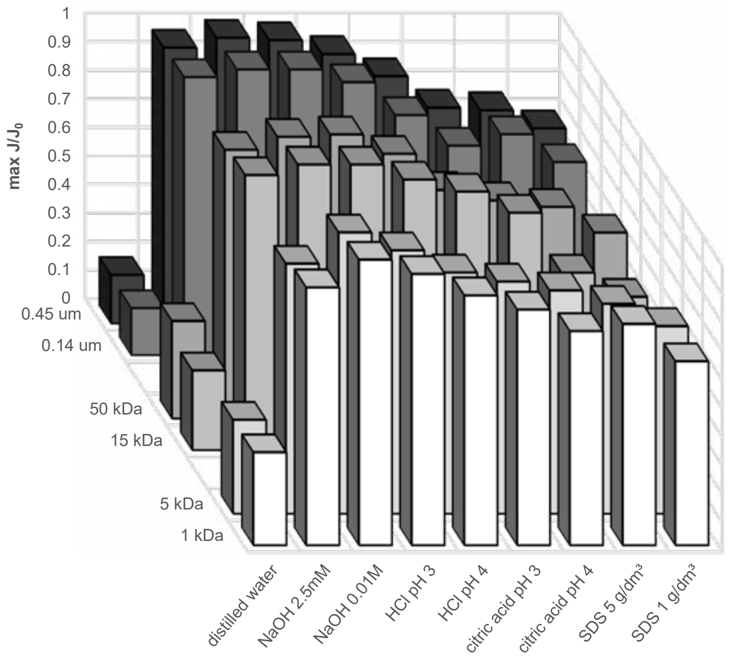

Fig 5. Effect of cleaning agent type on membrane regeneration after filtration of the liquid fraction of municipal-derived digestate, expressed as the ratio of the post-cleaning flux to the initial flux (J/J_o)

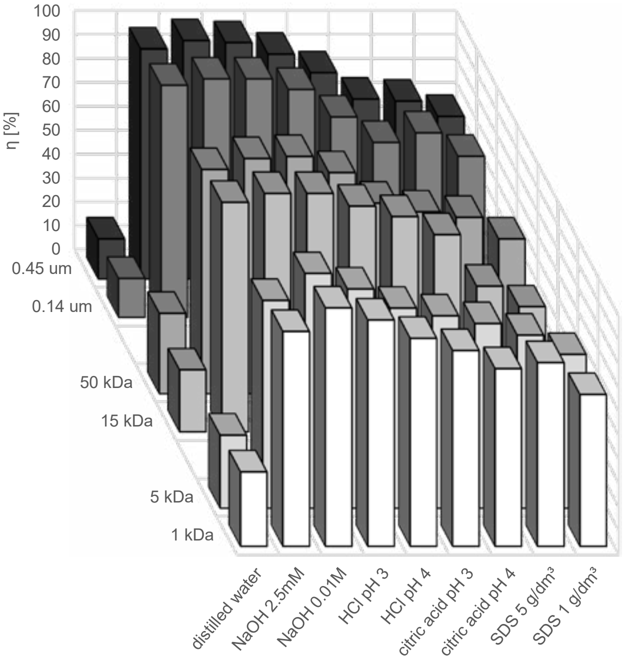

Fig 6. Comparison of membrane regeneration efficiency after 3 hours of cleaning with different agents for different membrane types

with larger pore sizes (MF). Surfactants (SDS at 1% and 5%) and distilled water demonstrated even lower efficiency, which confirms the limited effectiveness of physical or mild chemical cleaning against strongly bound organic-mineral deposits.

There is a clear trend indicating that membranes with lower cut-offs (1 and 5 kDa) are much easier to regenerate, regardless of the agent used. This may be due to a lower proportion of sediment penetrating deeply into the porous structure.

Therefore, it is reasonable to conclude that the efficiency of membrane regeneration depends significantly on the choice of chemical agent, its concentration, and the characteristics of the membrane. Cleaning agents at moderate concentrations of alkaline or acidic solutions provide the highest cleaning efficiency, making them the preferred choice for maintaining the efficiency of membrane processes, particularly in applications involving the liquid fraction of digestate.

Analysis of the time required to recover 90% of the maximum purification efficiency, which was achieved after 180 minutes of regeneration, revealed significant differences among the cleaning agents and membrane types tested

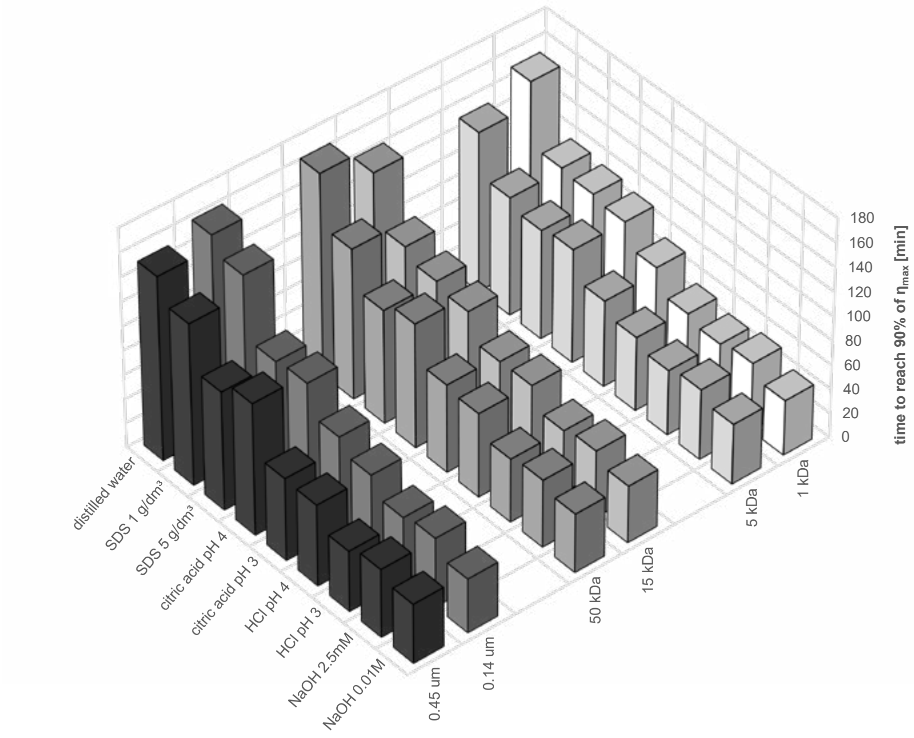

Fig 7. Treatment process kinetics: time required to reach 90% η_{max} for membranes fouled with liquid fraction of municipal digestate

(Figure 7). The most effective chemicals - particularly a 0.01M NaOH solution and HCl solution at pH 3 - allowed the expected level of regeneration to be achieved in just 45 to 55 minutes, regardless of membrane type. These short regeneration times combined with high efficacy suggest that these agents rapidly affect the main components of the sludge. This may indicate that the predominant fouling mechanisms are susceptible to degradation in either alkaline or acidic environments.

Significantly extended recovery times exceeding 100 minutes were observed for less aggressive agents, such as citric acid at pH 4 and SDS at 1 g/dm³. Physical cleaning with distilled water was particularly inefficient, requiring longer membrane contact times to reach the 90% flux recovery threshold. This confirms the limited effectiveness of non-chemical methods.

The results demonstrate the importance of selecting the appropriate cleaning agent based not only on its final efficiency but also on its dynamic performance. This consideration is crucial for optimizing membrane system downtime. Reducing regeneration time without sacrificing cleaning efficiency enables continuous operation and reduces the operating costs of membrane systems.

Conclusions

The study revealed that the porous structure of flat ceramic membranes significantly affects their transport properties, susceptibility to fouling, and regeneration efficiency. Membranes with larger pore diameters exhibited lower initial hydraulic resistance and greater susceptibility to reversible surface fouling. Conversely, despite higher hydraulic resistance, membranes with smaller cut-off showed greater resistance to permeate flux decline and higher separation efficiency for fine particles over longer operating times.

Analysis of fouling mechanisms confirmed that organic fouling was the predominant contributor, leading to either surface fouling or penetration depending on the membrane pore size and characteristics. The effectiveness of chemical cleaning depended strongly on the cleaning agent; the best results were obtained using mild solutions of NaOH (0.01M) and HCl at pH 3. These agents allowed for nearly complete membrane recovery within a relatively short time (less than 60 minutes). In contrast, milder chemicals were significantly less effective, particularly for microporous membranes.

The study's conclusions clearly indicate that:

- medium cut-off membranes (5 and 15 kDa) represent an optimal compromise between separation efficiency, fouling resistance, and regeneration effectiveness;
- in practical applications, membrane selection should account for separation requirements, susceptibility to fouling, and the feasibility of effective cleaning;
- a regeneration strategy employing moderately concentrated alkaline and acidic agents enables membrane system to maintain high operational efficiency while reducing downtime and operating costs;
- fouling mechanisms and their removal efficiency are closely linked to membrane characteristics, necessitating a customized approach to the design and operation of membrane systems.

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Skuteczność środków czyszczących w usuwaniu zanieczyszczeń z membran ceramicznych po filtracji ciekłej frakcji pofermentu pochodzenia komunalnego

Streszczenie. Celem niniejszej pracy była ocena skuteczności wybranych środków chemicznych do czyszczenia płaskich membran ceramicznych zanieczyszczonych podczas filtracji ciekłej frakcji pofermentu z biogazowni komunalnej. Badania skupiały się na tym, jak struktura porów membrany wpływa na właściwości transportowe, mechanizmy zanieczyszczania i wydajność regeneracji, aby określić optymalne strategie czyszczenia. Zastosowano sześć rodzajów płaskich membran ceramicznych o różnych wartościach cut-off (1 kDa – 0,45 µm). Ciekła frakcja pofermentu została przefiltrowana w systemie dead-end przy stałym ciśnieniu 0,2 MPa. Zanieczyszczone membrany zostały oczyszczone za pomocą wodorotlenku sodu (NaOH), kwasu solnego (HCl), kwasu cytrynowego i dodecylosiarczanu sodu (SDS) w różnych stężeniach, a także za pomocą wody redestylowanej. Skuteczność czyszczenia określono poprzez pomiar odzysku początkowego strumienia permeatu. Zanieczyszczenie membrany następowało szybko, a główną przyczyną były substancje organiczne. Najskuteczniejsze czyszczenie uzyskano przy użyciu roztworu NaOH o stężeniu 0,01 M i roztworu HCl o pH 3, które przywróciły prawie 100% początkowego przepływu dla większości membran w czasie krótszym niż 60 minut. Słabsze środki i woda redestylowana były znacznie mniej skuteczne. Membrany ultrafiltracyjne o średniej wielkości cząsteczek (5 i 15 kDa) wykazały najlepszą równowagę między wydajnością separacji, odpornością na zanieczyszczenia i łatwością regeneracji. Wybór zarówno membrany, jak i środka czyszczącego ma kluczowe znaczenie dla wydajnej pracy. Membrany ultrafiltracyjne o średnim cut-off (5–15 kDa) stanowią optymalne rozwiązanie. Strategia regeneracji z wykorzystaniem umiarkowanie stężonych roztworów alkalicznych i kwasowych zapewnia wysoką wydajność operacyjną i minimalizuje przestoje instalacji membranowych.